Abstract No. Jørg0396

Time Resolved X-Ray Diffraction Studies of the Hydrothermal Synthesis of ZnSe

J.-E. Jørgensen , T. R. Jensen, A. N. Christensen, (Dept. of Chem., Univ. of Aarhus, Denmark) and J. C. Hanson (Chem. Dept., BNL)

Beamline(s): X7B

Introduction: ZnSe is a II-VI semiconducting compound with a large direct band gap and it is used for infrared windows and lenses. Several methods such as molecular beam epitaxi, metalorganic chemical vapor deposition and organometallic vapor phase epitaxy have been developed for the synthesis of nanoscale particles of this compound. Recently nanocrystalline ZnSe was synthesized direct form the element under hydrothermal conditions using water as the reaction medium [1]. We have performed an *in situ* X-ray powder diffraction study the synthesis of ZnSe under hydrothermal conditions with the aim of elucidating the reaction path for the formation of this compound.

Methods and Materials: A 1:1 mixture of Zn and Se powders was loaded into a capillary together with water. The capillary tube was pressurized (max. 25 bar) with nitrogen gas to prevent boiling of the water. Powder patterns were recorded by the use of a MAR image plate detector. The exposure time (30 sec.) plus the read-out-time for the image plate detector was 82 seconds per pattern. in the temperature range 170 to 200 ˚C with a counting time of 30 sec. per pattern. The selected wavelength and the detector-sample distance was refined using a powder sample of LaB_6 to λ= 0.90371(3) Å and 158.1 mm, corresponding to $(\sin\theta)/\lambda = 0.49$ Å^{-1}. In a typical experiment , the temperature was increased by two degrees per minute to a final temperature in the range 180 to 200˚C at which it was kept for two to three hours.

Results: The experiment shows that Zn powder initially reacts with water giving $Zn(OH)_2$ and H_2. Se is not participating in the reaction before the temperature is about 180 to 190 ˚C where the Se Bragg peaks disappear. Se is presumably starting to dissolve as a sol at this temperature. The disappearance of the Se Bragg peaks is seen to coincide with the onset of the formation of ZnSe. It is therefore concluded that H_2 reduces the dissolved Se to Se^{2-} which reacts with Zn^{2+} to form ZnSe. A typical stack of powder patterns is shown in the figure below.

Acknowledgments: The work at BNL was supported by contract DE-AC02-98CH10086 with the US DOE Div. of Chemical Sciences and the Danish National Research Council (Dansync).

References:

[1] Q. Peng, Y. Dong, Z. Deng, X Sun and Y. Li, Inorg. Chem. 2001, **40**, 3840-3841.